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14. ABSTRACT

California State University, Northridge (CSUN) received a grat of $199,500 from the DoD to complement its surface science system at the Materials Science and Engineering Research Center (MSERC). The improvement of our facilities and capabilities has attracted institutions and industrial partners who are interested in initiating exchange programs and joint research projects for engineering applications. This has greatly enhanced the quality of our educational programs in terms of technical projects and increased funding. These complex technological resources have helped us to remain competitive in a field that is constantly developing new concepts. In this endeavor, a fundamental component to the research was the involmement and education of students. MSER hired promising undergraduate students to work on research that required technical science equipment. Our research objectives included 1)the invetigation of the processing/structure/property relationships in nanoscale materials; 2) the development of accurate models to explain the underlying physical mechanisms responsible.

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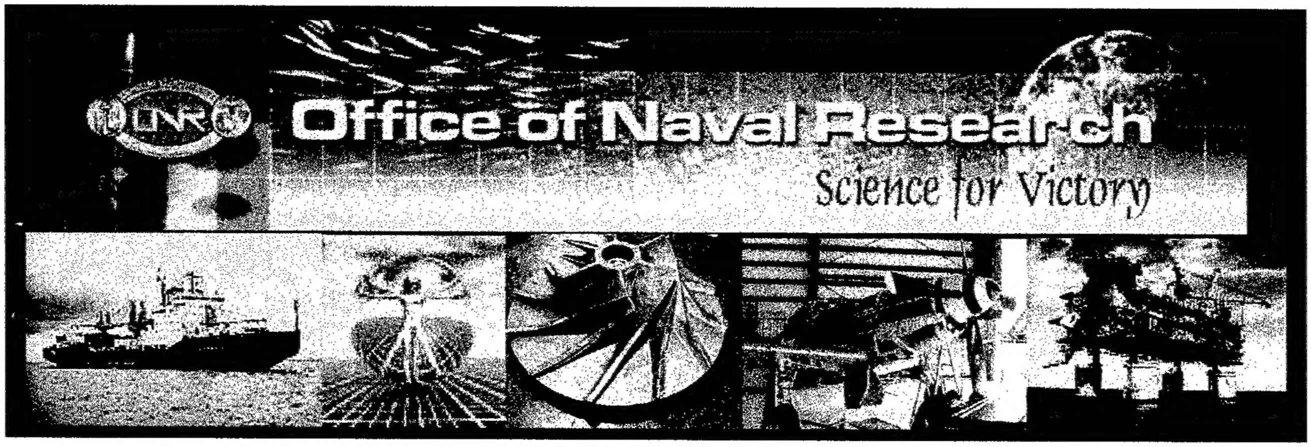

Acquisition of Secondary Ion Mass Spectrometer with Fracture Stage

Office of Naval Research
Contract # F49620-02-1-0022

Professor Behzad Bavarian
California State University, Northridge
December 2002

California State University
Northridge

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Executive Summary

California State University, Northridge (CSUN) received a grant of $199,500 from the DOD ONR to complement its surface science system at the Materials Science and Engineering Research Center (MSERC). The improvement of our facilities and capabilities has attracted institutions and industrial partners who are interested in initiating exchange programs and joint research projects for engineering applications. This has greatly enhanced the quality of our educational programs in terms of technical projects and increased funding. These complex technological resources have helped us to remain competitive in a field that is constantly developing new concepts. In this endeavor, a fundamental component to the research was the involvement and education of students. MSERC hired promising undergraduate students to work on research that required technical science equipment. Our research objectives included 1) the investigation of the processing/structure/property relationships in nanoscale materials; 2) the development of accurate models to explain (from an atomistic level) the underlying physical mechanisms responsible for observed behavior; and 3) the experimental validation of the models. The equipment of Physical Electronics, Kratos Analytical Inc. and VG Scientific manufacturers of the dynamic SIMS and in-situ fracture stage were evaluated to determine best value. Since the Kratos Ultra Axis Multichannel Spectroscope had already been selected for the main platform, the dynamic SIMS and in-situ fracture stage were also purchased from Kratos Analytical Inc. This choice would facilitate installation and minimize interface complications with the Multichannel Spectroscope system.

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Final Report

California State University, Northridge (CSUN) received a grant of $199,500 from the DOD ONR to complement its surface science system at the Materials Science and Engineering Research Center (MSERC). The improvement of our facilities and capabilities has attracted institutions and industrial partners who are interested in initiating exchange programs and joint research projects for engineering applications. This has greatly enhanced the quality of our educational programs in terms of technical projects and increased funding. These complex technological resources have helped us to remain competitive in a field that is constantly developing new concepts. In this endeavor, a fundamental component to the research was the involvement and education of students. MSERC hired promising undergraduate students to work on research that required technical science equipment. Our research objectives included 1) the investigation of the processing/structure/property relationships in nanoscale materials; 2) the development of accurate models to explain (from an atomistic level) the underlying physical mechanisms responsible for observed behavior; and 3) the experimental validation of the models.

CSUN requested funding from the Keck Foundation to purchase a multichannel spectroscope, Auger Electron Spectroscopy, X-ray Photoelectron Spectroscopy and Secondary Ion Mass Spectroscopy (AES, XPS, & SIMS), but received partial funding. The amount was not sufficient to include the dynamic SIMS detector, a crucial component for analyzing and characterizing MEMS-based sensors and substrates, thin film coatings, corrosion resistant coatings, polymer composite materials and nanostructural materials used in military and space applications.

Elemental characterization of defects and contamination on surfaces is a necessity for failure analysis. Energy Dispersive X-ray (EDS) combined with Scanning Electron Microscopy (SEM) provide a rapid and effective tool for characterizing particles and defects for samples 1 micron and larger. For submicron particles and defects, the ideal tool is an Auger Electron Spectroscopy with XPS & SIMS. With these instruments, features of several hundred angstroms and smaller can be characterized. For probing, SIMS uses a beam of ions to strike the surface and knock off atoms of the sample material. These atoms are ionized, identified and measured using mass spectrometry. SIMS helps test theories and confirms implementations in the development of new applications. SIMS techniques complement photoluminescence and electrical measurements by providing depth distribution information with excellent detection sensitivity.

In Summary, the addition of a SIMS detector provided MSERC with highly advanced analysis capabilities and the opportunity to further expand the educational opportunities of our students and faculty. MSERC currently has 12-15 underrepresented minority undergraduate students each year and 4 graduate students involved in various research projects funded by NASA/JPL, Hughes Research Lab, and the Cortec Corporation. This equipment also has direct application to the subject matter of several engineering materials science courses; exposure to it provides hands on training and a better awareness and understanding of surface science techniques than could be obtained from a lecture.

Quadrupole Mass Analyzer, SIMS Specifications

Secondary Ion Mass Spectrometry (SIMS) is a very sensitive surface and thin film analysis technique used for characterizing trace and major elements on solid surfaces. SIMS uses an ion beam of 200 eV to several KeV in energy to sputter a sample surface. The portion of sputtered material that is ionized is accelerated into a mass spectrometer and identified. The positive or negative secondary ion count of each monitored element is recorded with the time sputtered for subsequent conversion into a concentration versus depth profile. The SIMS technique can handle a full range of materials including Si, SiO2, BPSG, SiNx, SiOxNy, Al(SiCu), TiNx, TiW, silicides, GaAs, AlGaAs, GaN, InP, C(DLC), ITO, MCT/CT, etc.

General capabilities of SIMS:

- Detection of all elements from hydrogen through uranium.
- Trace element analysis with 1e12 - 1e15 at/cc (0.1 ppb - 0.1 ppm) lower detection limit for most elements.
- Quantitative with standards (10% typical accuracy) and semi-quantitative using relative tabulated sensitivity factors.
- Precision < 2 %.
- Depth resolution: 10-100 Angstroms
- Monolayer depth information.
- Elemental imaging of < 250 um area with 1 μm lateral resolution.
- Isotope abundance determination.
- Small area analysis (<25 um).

The composition of the outer most atomic layers of a material plays a critical role in properties such as: chemical activity, adhesion, wettability, electrostatic behavior, corrosion resistance and biocompatibility. In addition, contaminants, process residues, diffusion products, and impurities are typically present at the surface of a solid sample. The ability to analyze thin film structures, via sputter depth profiling, provides the unique opportunity to characterize materials used in thin films and to study their interaction with materials in adjacent layers. Surface analysis instruments are often used to guide the development of deposition processes and to validate a process when new deposition equipment is installed. The ability to analyze submicron defects or particulate contaminants is of critical importance to increasing product yield in a number of industrial applications. Some of the research areas that can benefit from SIMS are:

Microelectronics -- The small dimensions of structures within microelectronic devices make surface sensitive analytical techniques ideal for composition and thickness of deposited layers, defect characterization, particle identification, and the presence of process residues. The vast majority of microelectronic devices manufactured for military and aerospace applications are subjected to variable and often severe environments. These hostile environments, including ambient relative humidity combined with elevated temperatures, and the presence of aggressive species, high dosage of γ-ray radiation and extremely long duration, are responsible for the principal failures (e.g., metallization failures, electromigration, metallic interface failures, silicon oxide failures, and metal/oxide interface

failures) in electronic packages. Microscopically, these failures are caused by oxidation, corrosion, defect creation and diffusion, interfacial segregation and interdiffusion and mechanical malfunction. The synthesis, processing, properties, and limits of performance of these materials in natural or radiation environments, the extreme temperatures and pressures associated with the space environment will be investigated.

Corrosion -- Discoloration on metal surfaces is often the result of variations in oxide thickness that can be caused by corrosion. Surface analysis can be used to determine the thickness and composition of oxide layers and can often identify corrosive elements such as chlorine in corroded areas. The corrosion resistance of stainless steel is the result of a chromium oxide that forms on the surface protecting the more reactive iron. The thickness and chemistry of this "passivation" layer can be examined with surface analysis techniques to help ensure proper surface preparation for the best corrosion resistance. Development of new and environmentally acceptable coatings to prevent corrosion of marine vessel hulls, aircraft and weapons systems is possible with these advanced surface science techniques.

Coating Materials and MEMS based sensors-- Anti reflective coatings, hard coats, low emissivity coatings, and mirrors on glass or plastic substrates can be probed by surface analysis to determine: layer composition and thickness, presence of adhesion layers, and composition of surface and buried defects.

Biotechnology -- Metallic and polymeric implantable devices are routinely characterized by surface analysis methods to study the effects of surface chemistry on biocompatibility and device performance. Utilizing MEMS technology, smart drug delivery systems can be developed.

Failure Analysis -- Surface analysis can prove invaluable for certain types of failure analysis including: adhesion failure resulting from surface contamination, particle identification, brittle fracture due to grain boundary impurities. It can also improve the life expectancy and reliability of materials.

Specifications of Quadrupole SIMS Analyzer

A standard Hiden quadrupole mass analyzer SIMS attachment and the ion gun used for sample cleaning and depth profiling was installed on the multi channel system. The quadrupole mass spectrometer is able to detect masses ranging from 0-511 amu (Figure 2). This module contains necessary power supplies for quasi-simultaneous operation of all analysis modes. The system operates in either positive or negative ion mode and has its own PC system with software to control the SIMS instrument, data acquisition and data processing. SIMS specification and resolution performance for positive ions: 50,000 cps per nA primary beam on the $27Al^+$ plus 27 AlO^+ peaks using 5keV Argon ions. Negative ions: 10,000 cps per nA primary beam on KBr^- using 5keV Argon ions, and Mass Range: 0-511 amu, 500 Dalton Hiden system.

Ion Etching Source: The ion gun system has an energy range of 0.2-5 keV and is differentially pumped. Two refillable mini Ar Gas cylinders (one spare cylinder) were included with the system and rastering of the beam is a standard feature. Pressure in the analysis chamber during etching is normally maintained at less than 5×10^{-7} torr. The spot

size of the ion beam is variable down to 150 μm and the maximum beam current is about 10uA at 5 kV.

Fracture Stage: This system is equipped with a fracture stage that facilitates the fracturing of samples in an ultra high vacuum (UHV) environment and subsequent examination by XPS, AES/SAM or SIMS. Both impact and slow fracture modes are included on this system (Figure 3).

Sources for the dynamic SIMS and in-situ fracture stage:

Physical Electronics
Dr. Jude Koenig
17121 Parkview Drive
Morgan Hill, CA 95037
Phone: (408) 776-1796
Fax: (408) 776-7427
Web page: www.phi.com

Kratos Analytical Inc.
Dr. W. Alan Wolstenholme
13840 West Oak Glen Road
Valley Center, CA 92082
Phone: (760) 749-9306
Fax: (760) 749-6599
Web page: www.kratos.com

VG Scientific
Christopher Macey
100 Cummings Center
Suite 435-G
Beverly MA 01915
Phone: (978) 921 9393
Fax: (978) 921 9366
www.vgscientific.com

The dynamic SIMS and in-situ fracture stage equipment from these manufacturers was evaluated. The Kratos Ultra Axis Multichannel Spectroscope had already been selected for the main platform (Figure 1); therefore the dynamic SIMS and in-situ fracture stage were also purchased from Kratos Analytical Inc. This choice was made to facilitate installation and minimize interface complications with the Multichannel Spectroscope system.

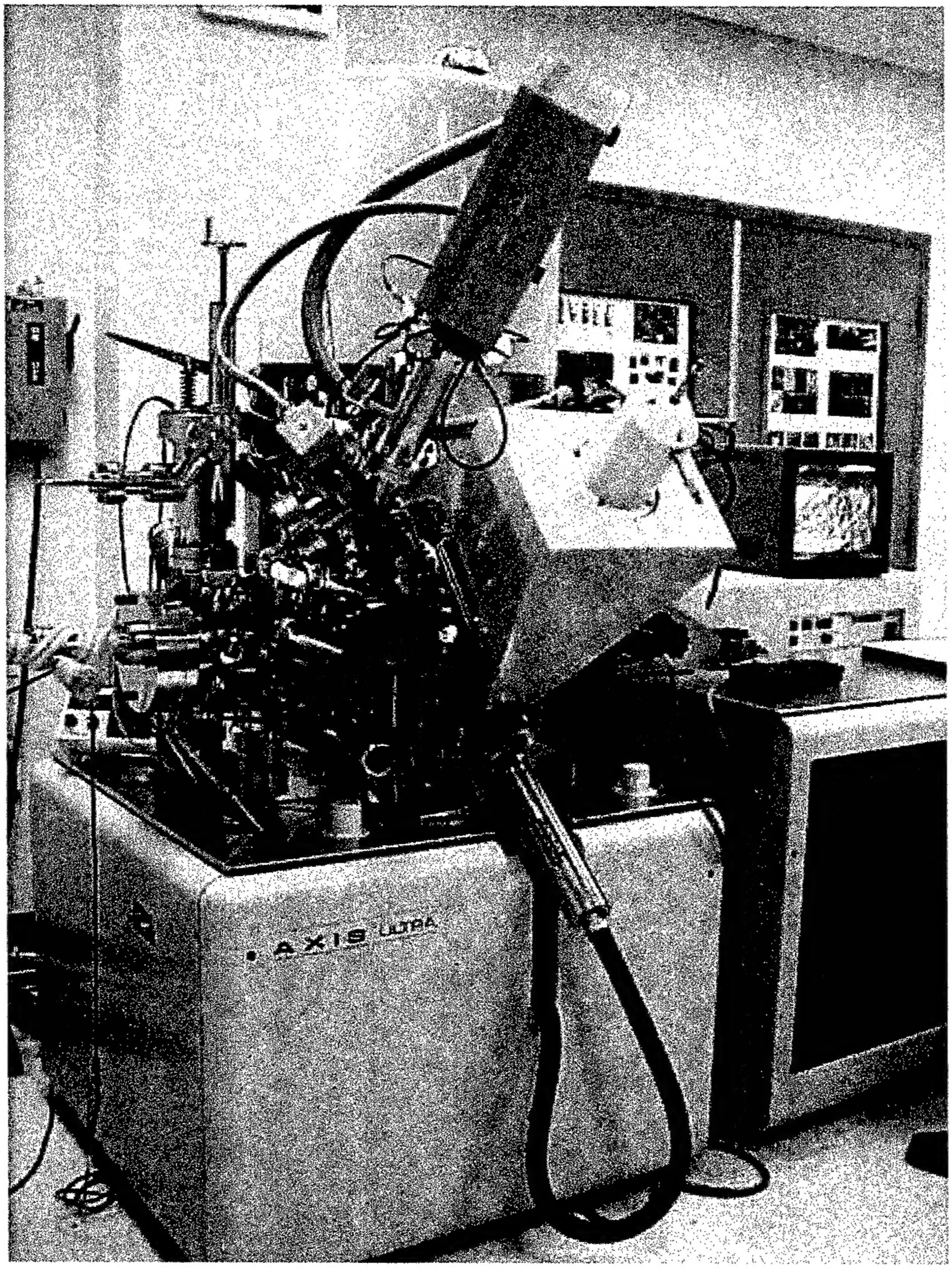

Figure 1: The Kratos Ultra Axis Multichannel Spectroscope is fully installed and in operation since June 2002. Quadrupole SIMS and In-situ fracture stage were also installed on this system.

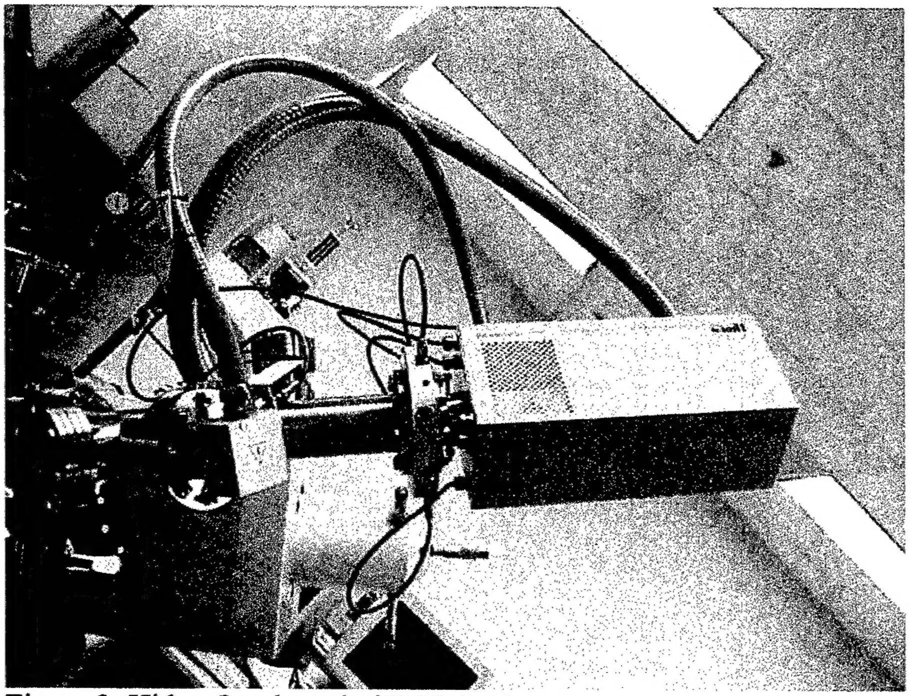

Figure 2: Hiden Quadrupole SIMS Analyzer

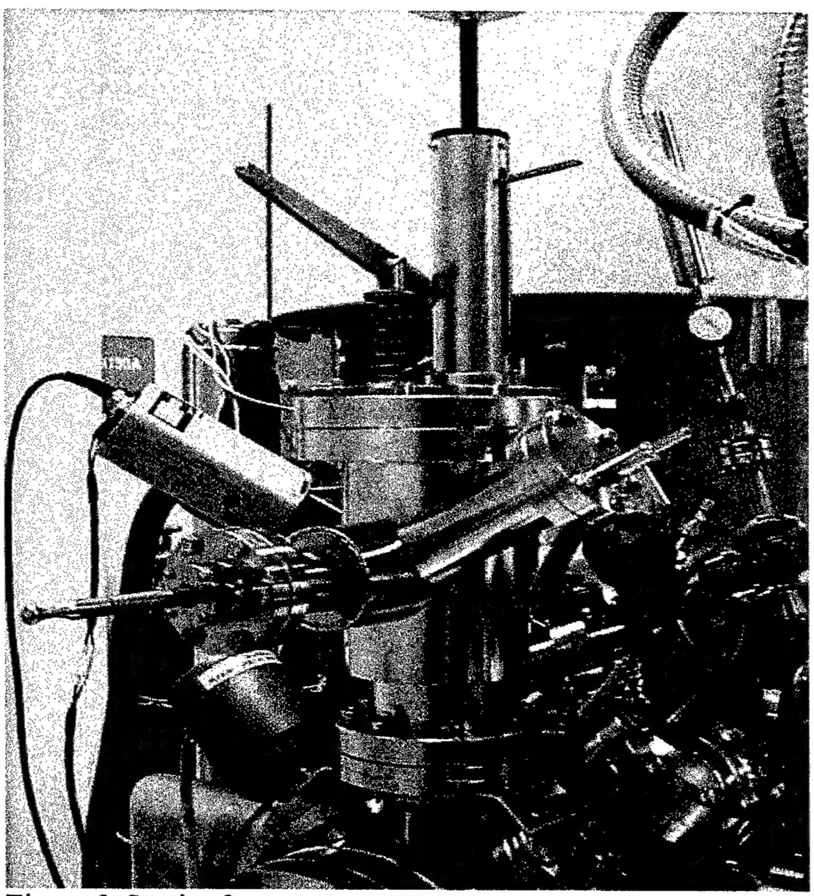

Figure 3: In-situ fracture stage

Impact of the Instrumentation on Education and Research

MSERC currently has 12-15 underrepresented minority undergraduate students each year and 4 graduate students involved in various research projects funded by NASA/JPL, Hughes Research Lab, and the Cortec Corporation. Roughly 25 students (seniors and graduate level) will be using this equipment in MSE 550-Thin Film Technology, MSE 531-Corrosion, MSE 624-Failure Analysis, MSE 647-Nonoptical Microscopy & Spectroscopy and MSE 630-Electronic Materials. The Advanced Materials and Corrosion Laboratories of MSERC provide a state-of-the-art facility for materials characterization research. It is well equipped with tools to perform detailed studies and investigations of the structure, chemical and physical properties, microstructure and nanostructure. Some of the equipment represents the latest available technology in the industry for the performance of energy dispersive microchemical analysis, atomic force nanoscope, 3-D C-Scan using ultrasonic detector, and scanning reference electrode measurement. The facility provides excellent opportunities for performing highly advanced research studies in the field of materials engineering. Among the research topic areas pursued by the materials engineering research program in recent years are:

- Failure Analysis, Prevention, and Reliability Modeling for Submicron Electronics Technology in Space Application, A Research partnership of CSUN with JPL/NASA, to study various failure mechanisms of submicron electronics devices, MEMS sensors and develop atomistic models for these failures. ($2 million, 2000-2004).
- Crevice Corrosion Behavior of Aluminum Alloys and its growth rate modeling for application in automotive industries. (HRL $120,000, 1999-2001)
- Metallurgical Characteristics of Aluminum Magnesium Nanophase alloys, to understand the mechanical properties of these materials for cryogenic and high temperature applications. (CSUN/Boeing $40,000, 1998-2000)
- Electrochemical AC Impedance Technique to Investigate the Inhibition Efficiency of Migrating Corrosion Inhibitors for Steel Rebar in Concrete. This investigation determined the corrosion behavior of steel rebar in concrete in high chloride environments through the use of electrochemical impedance spectroscopy to find a protective inhibition system. (MWD/Cortec $75,000, 1994-2000)
- Hydrogen Embrittlement of Beta 21S Ti-alloy, Hot Corrosion of Beta 21S Ti-alloy and Stress Corrosion Cracking of Beta 21S Ti-alloy, The Beta 21S is a very promising alloy, and its application in jet engines and spacecraft.
(NASA $110,000, 1992-1994)
- Corrosion behavior of (Ti_xAl, Fe_xAl and Nix_xAl) Intermetallic compounds. These intermetallic materials provide excellent strength to weight ratio, which make them very useful for spacecraft application. (NASA $110,000, 1988-1990)
- Effects of Atomic Oxygen on the Space-based Instruments in a Low Earth Orbit, to understand how different materials behave in the space environment.
(Lockheed Martin $45,000, 1998-1999)
- Environmental Effects on the Fatigue Crack Growth Rate of High Strength Low Alloy Structural Steel (HSLA80) proposed for future use in oil tankers and vessels. (NSF $125,000, 1993-1998)
- Strength and ductility of Reinforced Concrete Composite Cylinders to improve the concrete columns performance during earthquakes. (NSF $125,000, 1993-1998; PI: Dr. Shively)

- Electrochemical failure of microelectronics & Failure analysis of microelectronic devices in space. (Lockheed Martin $22,000, 1997)
- Corrosion of rebar in concrete. (CORTEC/VALBUNO $34,000, 1996-1994)
- Hydrogen Embrittlement of the High Strength Steels. (MWD $55,000, 1997-1998)
- Construction Weldment Failures due to 1994 Northridge Earthquake; Welding failures, Improper design and materials. (NIST $226,000, 1995-1996)
- Corrosion Fatigue Behavior of High Strength Structural Steels; Weldment, HAZ & base plate. (NSF $255,000, 1995-1998)

The principal investigator for all projects unless otherwise noted is Dr. Behzad Bavarian.

Budget and cost sharing

		Amount	
Multichannel Spectroscope (AES & XPS)		$ 555,000	Keck
[redacted]		[redacted]	[redacted]
XPS & SAM Sample Preparation Stage		$ 34,000	CSUN
[redacted]		[redacted]	[redacted]
Remodeling room for equipment installation		$ 50,000	CSUN
PI release time	(25% rate)	$ 57,000	CSUN
Student stipends	(12 students)	$ 126,000	NASA

Total requested from DOD is $199,500.00.

The Kratos Ultra Axis Multichannel Spectroscope was purchased for $589,000 (funded by the Keck Foundation and California State University Northridge). The dynamic SIMS and in-situ fracture stage was purchased for $199,623 from Kratos Analytical Inc. (funded by DOD and some minor contributions from the NASA-IRA grant.) This equipment came with a two year full service contract warranty and a third year that covers parts and labor. Remodeling and room preparation for equipment installation was funded by Cal State Northridge ($52,000). The principal investigator was released from his teaching assignment (25% of teaching load) to supervise installation and commissioning of the equipment ($57,400). The student stipends for the Undergraduate Research Program were provided by NASA-IRA NCC5-513 grant ($126,000).

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